FORM	PTO-139	00 (Modified) U.S. DEPARTMENT OF COMMERCE PATENT AND	TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER		
TRANSMITTAL LETTER TO THE UNITED STATES 220803US0PCT				220803US0PCT		
DESIGNATED/ELECTED OFFICE (DO/EO/US) U.S. APPLICATION NO (IF KNOWN, SEE 37 CFF						
	CONCERNING A FILING UNDER 35 U.S.C. 371					
INTE	RNAT	IONAL APPLICATION NO INTERNATIONAL FILIN		PRIORITY DATE CLAIMED		
anar r		PCT/EP00/09135 18 Septemb	per 2000	20 September 1999		
		NVENTION COLYTIC TRANSFORMATION OF ORGANIC (COMPOUNDS			
APPL	ICAN	T(S) FOR DO/EO/US		· · · · · · · · · · · · · · · · · · ·		
PUE	TTE	R Hermann et al.				
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Appl	icant l	nerewith submits to the United States Designated/Elected O	office (DO/EO/US) th	e following items and other information		
į 1.	\boxtimes	This is a FIRST submission of items concerning a filing	under 35 U.S C 371.			
2.		This is a SECOND or SUBSEQUENT submission of ite	_			
3.	\boxtimes	This is an express request to begin national examination p (9) and (24) indicated below.	procedures (35 U.S.C	371(f)). The submission must include itens (5), (6),		
4.	\boxtimes	The US has been elected by the expiration of 19 months f	rom the priority date	(Article 31)		
5.	\boxtimes	A copy of the International Application as filed (35 U.S C	C 371 (c) (2))			
		a. — is attached hereto (required only if not communication).	cated by the Internat	tional Bureau)		
		b. 🗵 has been communicated by the International Bureau.				
		c. Is not required, as the application was filed in the United States Receiving Office (RO/US).				
6.	\boxtimes	An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).				
		a. 🖾 is attached hereto.				
7	1527	b. has been previously submitted under 35 U.S.C. 154(d)(4).				
7.	\boxtimes	Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C 371 (c)(3))				
		a. □ are attached hereto (required only if not communicated by the International Bureau) b. □ have been communicated by the International Bureau.				
		c. \(\square\) have not been made; however, the time limit for making such amendments has NOT expired.				
		d. \(\text{ have not been made and will not be made} \)				
8.		An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).				
9.	\boxtimes	An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).				
10.		An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U S.C. 371 (c)(5)).				
11.		A copy of the International Preliminary Examination Report (PCT/IPEA/409)				
12.	\boxtimes	A copy of the International Search Report (PCT/ISA/210)				
It	ems 1	3 to 20 below concern document(s) or information inclu	ded:			
13.	\boxtimes	An Information Disclosure Statement under 37 CFR 1.97				
14.		An assignment document for recording. A separate cover	sheet in compliance	with 37 CFR 3 28 and 3.31 is included		
15.		A FIRST preliminary amendment.				
16.		A SECOND or SUBSEQUENT preliminary amendment.				
17. 18.		A substitute specification A change of power of atterney and/or address letter				
19.		A change of power of attorney and/or address letter. A computer-readable form of the sequence listing in accordance with PCT Rule 13ter 2 and 35 U S.C. 1 821 - 1 825				
20.		A second copy of the published international application under 35 U.S.C. 154(d)(4)				
21.		A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).				
22.		Certificate of Mailing by Express Mail				
23.	\boxtimes	Other items or information:				
		Notice of Priority Form PTO-1449 PCT/IB/304		A.,		

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24.	The fol	lowing fees are sub					CA	LCULATIONS	PTO USE ONLY
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	and all claims satisfied provisions of PCT Article 33(1)-(4)								
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b.	to gover the shows fees								
c. The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 15-0030 A duplicate copy of this sheet is enclosed.									
d. Fees are to be charged to a credit card. WARNING: Information on this form may become public Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.									
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR									
1.137(a) or (b)) must be filed and granted to restore the application to pending status. SEND ALL CORRESPONDENCE TO:									
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				Norman F. Oblon NAME 24,618					
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220803US-0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

HERMANN PUETTER ET AL

: ATTN: APPLICATION DIVISION

SERIAL NO: NEW U.S. PCT APPLN

(Based on PCT/EP00/09135)

FILED: HEREWITH

FOR: ELECTROLYTIC TRANSFORMATION:

OF ORGANIC COMPOUNDS

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows:

IN THE CLAIMS

Please cancel Claims 1-9.

Please add the following new claims.

- 10. (New) A process for the electrolytic transformation of at least one organic compound in an electrolysis cell, wherein the organic compound is both oxidized and reduced at one electrode.
- 11. (New) The process as claimed in claim 10, wherein the organic compound is both oxidized and reduced at the anode.

- 12. (New) The process as claimed in claim 11, wherein the organic compound is reduced by hydrogenation.
- 13. (New) The process as claimed in claim 11, wherein the anode is in contact with at least one hydrogenation catalyst.
- 14. (New) The process as claimed in claim 13, wherein the hydrogenation catalyst is a noble metal.
- 15. (New) The process as claimed in claim 13, wherein the hydrogenation catalyst is applied to a graphite felt.
- 16. (New) The process as claimed in claim 13, wherein the hydrogenation catalyst is deposited on the anode from suspension.
- 17. (New) The process as claimed in claim 13, wherein the hydrogenation catalyst in the form of a suspension is brought into contact with the anode.
- 18. (New) The process as claimed in claim 10, wherein the organic compound is both reduced and oxidized at the cathode.
- 19. (New) The process as claimed in claim 10, wherein the electrode is a gas diffusion electrode.
- 20. (New) The process as claimed in claim 10, wherein the organic compound is furan or a furan derivative or furan and a furan derivative.

REMARKS

Claims 10-20 are active in the present application. Claims 1-9 have been cancelled. Claims 10-20 are new claims. Support for the new claims is found in the original claims. No new matter is believed to have been added by this amendment. An action on the merits and allowance of claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C.

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220803US-0PCT

Marked-Up Copy
Serial No:

Amendment Filed on: 3-20-2002

IN THE CLAIMS

Claims 1-9 (Cancelled).

Claims 10-20 (New).

AS ORIGINALLY FILED

Electrolytic transformation of organic compounds

The present invention relates to a process for the electrolytic transformation of organic compounds, in which one electrode simultaneously serves to transfer both oxidation and reduction equivalents.

An objective of preparative organic electrochemistry is 10 to utilize the processes occurring in an electrochemical process at both electrodes in parallel.

is the oxidative such process An example of a dimerization of 2,6-dimethylphenol which is coupled with the dimerization of maleic esters (M.M. Baizer, 15 Baizer (editors), Μ. Μ. Η. Lund, Electrochemistry, Marcel Dekker, New York, 1991, pages 142 ff.).

20 A further example is the coupled synthesis of phthalide and t-butylbenzaldehyde, as described in DE 196 18 854.

However, it is also possible to utilize the cathode process and the anode process to prepare a single product or to destroy one starting material. Examples of such electrochemical processes are the production of butyric acid (Y. Chen, T. Chou, J. Chin. Inst. Chem. Eng. 27 (1996) pages 337-345), the anodic dissolution of iron which is coupled with the cathodic formation of ferrocene (T. Iwasaki et al., J. Org. Chem. 47 (1982) pages 3799 ff.) or the decomposition of phenol (A.P. Tomilov et al., Elektrokhimiya 10 (1982) page 239).

A new opportunity opens up when oxidation and reduction take place at one and the same electrode. This means that a substrate receives both oxidation and reduction equivalents either simultaneously or successively.

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- A successive transfer of oxidation and reduction equivalents at one electrode is possible, for example, in cyclic voltametry in which the potential of the electrode switches between positive and negative values at a predetermined rate within a period of time (cf., for example, D. Sawyer, A. Sobkowiak, J. Roberts Jr., Electrochemistry for Chemists, Second Ed., pages 68-78, John Wiley & Sons, Inc. New York 1995).
- 15 In the context of the present invention, it has now been found that an anode is able to transfer reduction equivalents to a substrate which has already taken up anodic redox equivalents.
 - 20 The process is not restricted to the anode, but can likewise be carried out at the cathode under suitable conditions.
 - It is an object of the present invention to provide an electrochemical process in which an organic compound is oxidized in one electrode process and the oxidation product is reduced at the same electrode.
 - We have found that this object is achieved by the process of the present invention for the electrolytic transformation of at least one organic compound in an electrolysis cell, wherein the organic compound is both oxidized and reduced at one electrode.
 - 35 In a preferred embodiment of the invention, the process of the present invention occurs in an undivided cell.

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In a further preferred embodiment of the invention, the organic compound is both oxidized and reduced, preferably hydrogenated, at the anode.

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In one preferred embodiment of the invention, the organic compound is hydrogenated by means of hydrogen at the one electrode, with hydrogen being formed as product at the other electrode or being supplied from outside to the electrolysis circuit.

In another preferred embodiment of the invention, the organic compound is both reduced and oxidized, preferably oxygenated, at the cathode. In the following, the invention will be illustrated by the example of anodes which simultaneously oxidize and hydrogenate.

Organic compounds which can be used as starting
materials in the process of the present invention are
in principle all organic compounds which have reducible
groups, preferably a furan or a substituted furan.

The process is not restricted to furan or substituted furans, but extends to all compounds and classes of compounds which are oxidizable or reducible or both by methods of organic electrochemistry. An overview of the classes of compounds is given by H. Lund, M. M. Baizer, (editors) "Organic Electrochemistry", 3rd edition, Marcel Dekker, New York 1991.

Suitable compounds of the stated classes are, for example, compounds containing double bonds, e.g.

35 1) Olefins:

where R_1 to R_4 are each an alkyl, aryl or alkoxy group, a hydrogen atom, a (substituted) amino group, a halogen atom or a cyano group and the substituents R_1 to R_4 may be identical or different.

The double bonds can be part of open-chain or cyclic compounds, and can be part of the ring or of the chain or of both.

For the purposes of the present invention, cyclic systems containing double bonds can be, in particular, aromatic systems.

In the compounds having a cyclic structure, one or more element(s) of the cyclic structure can be an unsubstituted or substituted heteroatom such as N, S, O, P.

The cyclic compounds may bear one or more functional substituents of the following types:

carboxyl groups, carbonyl groups (and N analogues),
25 carboxymethyl groups, nitrile groups, isonitrile
groups, azo (azoxy) groups, nitro groups, amino groups,
substituted amino groups, halogens.

2) Alkynes

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$$R_5 - \equiv -R_6$$

where R_5 and R_6 are each a hydrogen atom or an aryl, alkyl, carboxyl or alkoxycarbonyl group, and the substituents R_5 and R_6 may be identical or different.

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3) Carbonyl compounds

1.

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$$R_7$$
— CO — R_8

- 5 where R_7 and R_8 are each an aryl, alkyl, alkoxy or aryloxy group or a substituted amino group or a halogen atom, and the substituents R_7 and R_8 may be identical or different.
- In a preferred embodiment of the process of the present invention, furan is used. Apart from furan, substituted furans such as the following compounds are also preferred:
- furfural (furan 2-aldehyde), alkyl-substituted furans,

 furans bearing -CHO, -COOH, -COOR groups, where R is an
 alkyl, benzyl, aryl or, in particular, a C₁-C₄ alkyl
 group, -CH(OR₁)(OR₂) groups, where R₁ and R₂ may be
 identical or different and R₁ and R₂ are each an alkyl,
 benzyl, aryl or, in particular, C₁-C₄-alkyl group, and
 -CN groups in the 2, 3, 4 or 5 positions.

In the reaction of organic compounds according to the present invention, it is possible to use solvents and electrolyte salts as are described in H. Lund, M. M. Baizer, (editors) "Organic Electrochemistry", 3rd edition, Marcel Dekker, New York 1991.

According to the present invention, the oxidation of furans is preferably carried out in the presence of 30 methanol or in the presence of ethanol or a mixture thereof, but more preferably in the presence of methanol. These substrates can simultaneously be a reactant and solvent.

35 As solvents in the reaction of furans, it is generally possible to use all suitable alcohols in addition to

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the organic compound and the compound used for oxidation.

As electrolyte salts in the reaction of furans in the process of the present invention, it is possible to use not only NaBr but also, for example, alkali metal halides and/or alkaline earth metal halides, with bromides, chlorides and iodides being conceivable as halides. Ammonium halides can likewise be used.

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Pressure and temperature can be matched to the conditions which are customary in catalytic hydrogenations.

- In a preferred embodiment of the process of the present invention, the reaction temperature T is < 50°C, preferably < 25°C, the pressure p is < 3 bar and the pH is in the neutral region.
 - In a preferred embodiment of the process of the present 20 invention, intermediates are introduced in addition to the starting materials which are introduced into the preferably undivided electrolysis cell. intermediate refers to the product or products which is/are obtained by the electrolytic oxidation according 25 to the present invention of the organic compound or compounds, in particular a furan or a substituted furan or a mixture of two or more thereof, and is therefore present in the electrolysis circuit. The concentration additional intermediates is set by 30 electrocatalytic electrochemical and customary parameters, for example current density and type and amount of catalyst, or the intermediate is added to the circuit.

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In respect of the specific choice of the material of the electrodes, there is no restriction in the process of the present invention, as long as the electrodes are suitable for the process as described above.

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Preference is given to using graphite anodes in the electrolysis cell.

As regards the geometry of the electrodes in the electrolysis cell, there are essentially no restrictions in the context of the present invention. Examples of preferred geometries are plane-parallel electrode arrangements and annular electrode arrangements.

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In a preferred embodiment of the invention, the anode is in contact with at least one hydrogenation catalyst. particularly preferred embodiment, Ιn а hydrogenation catalyst or catalysts is/are part of a diffusion electrode. In a further preferred embodiment of the invention, the anode is a graphite electrode coated with a noble metal in the form of plates, meshes felts. In another preferred of embodiment of the invention, the hydrogenation catalyst is in the form of a suspension in the electrolyte and is continually brought into contact with the anode. catalyst, the hydrogenation Here, catalytically active material, is pumped around the cell or is deposited on an appropriately structured anode from suspension. An electrode of the latter type is described, for example, in DE 196 20 861.

In the process of the present invention, an organic compound is reduced, preferably hydrogenated, at the anode by means of the hydrogen which is formed as product in the cathode process. This hydrogenation

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preferably takes place by the compound to be hydrogenated being brought into contact with one or more hydrogenation catalysts which are in turn brought into contact with the anode.

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As regards the choice of hydrogenation-active catalysts, there are in principle no restrictions for the purposes of the process of the present invention. All catalysts known from the prior art can be used. Examples which may be mentioned are the metals of transition groups I, II and VIII of the Periodic Table, in particular Co, Ni, Fe, Ru, Rh, Re, Pd, Pt, Os, Ir, Ag, Cu, Zn and Cd.

20 As, Bi or Sb.

It is naturally also possible for the hydrogenationactive materials described to comprise a mixture of two or more of the specified hydrogenation metals, which may be contaminated by, for example, one or more of the abovementioned elements.

Of course, it is also conceivable for the hydrogenation-active material to be applied to an inert support. As such support systems, it is possible to use, for example, activated carbon, graphite, carbon black, silicon carbide, aluminum oxide, silicon dioxide, titanium dioxide, zirconium dioxide, magnesium dioxide, zinc oxide or mixtures of two or more thereof, e.g. as a suspension or as fine granules.

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In a preferred embodiment of the present invention, the hydrogenation-active material is applied to a base material for gas diffusion electrodes.

- 5 The present invention accordingly also provides a process as described above in which the base material for gas diffusion electrodes is laden with a hydrogenation-active material.
- 10 Possible hydrogenation-active materials with which the gas diffusion electrode system is laden are all the above-described hydrogenation catalysts. Of course, it is also possible to use a mixture of two or more of these hydrogenation catalysts as hydrogenation-active material.

For the purposes of the process of the present invention, it is naturally also conceivable for the gas diffusion electrode material to be laden with hydrogenation-active material and for use to be made of additional hydrogenation-active material which is identical to or different from that with which the gas diffusion electrode material is laden.

- 25 Furthermore, the present invention provides, in general form, for the use of a gas diffusion electrode for the electrolytic transformation of an organic compound, preferably an unsaturated organic compound, in an electrolysis cell.
- The following examples illustrate the present invention.

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Example 1

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An undivided cell having 6 annular electrodes having a surface area per side of 15.7 cm² was used. The electrodes were separated from one another by 5 spacer meshes having a thickness of 0.7 mm.

The electrodes comprised graphite plates each having a thickness of 5 mm and having one side coated with gas diffusion electrode material. This material was in turn laden with 5.2 g of Pd/m^2 .

The gas diffusion electrode was made the cathode.

The electrolyte mixture consisted of 30 g of furan,

57.4 g of 2,5-dimethoxydihydrofuran, 2 g of NaBr and
110.6 g of methanol.

The electrolysis was carried out at 0.5 A and a temperature of about 17°C. The cell voltage rose from 20 14.6 V to 20.7 V. The electrolysis was followed by gas chromatography.

After 1 F/mol of furan, the GC-percent by area of furan had been reduced from 22.7% to 17.8%, while the proportion of dimethoxydihydrofuran remained constant at 31 percent by area. At the same time, 0.9% of 2,5-dimethoxytetrahydrofuran was formed.

This example shows that the cathode is capable of catalytic hydrogenation. When graphite plates alone are used, i.e. not in the presence of a hydrogenation catalyst, good yields of 2,5-dimethoxydihydrofuran are obtained in agreement with the literature (H. Lund, M. M. Baizer, Organic Electrochemistry, Marcel Dekker, New York, 1991, page 720); 2,5-dimethoxytetrahydrofuran is not disclosed and was not found.

Example 2

- Example 2 was carried out using the arrangement from Example 1, but here the anode was provided with electrocatalytically active material. Instead of a gas diffusion cathode, a gas diffusion electrode laden with 5.2 g of Pd/m² was used as anode.
- 10 The electrolyte mixture consisted of 30 g of furan, 57.4 g of 2,5-dimethoxydihydrofuran, 2 g of NaBr and 110.6 g of methanol.
- The electrolysis was carried out at 0.5 A and a 15 temperature of 17°C. The cell voltage rose from 16.3 V to 19.5 V. The electrolysis was followed by gas chromatography.
 - After 1 F/mol of furan, the GC-percentage by area of furan had been reduced from 22.7 to 16.9%, and the GC-percentage by area of 2,5-dimethoxydihydrofuran remained at 30%. At the same time, 3.3% of 2,5-dimethoxytetrahydrofuran were formed.
 - 25 The comparison shows that the anode operates even more effectively than the cathode. This arrangement is thus not purely dependent on the presence of catalytically active material in the cells.

We claim:

- 5 1. A process for the electrolytic transformation of at least one organic compound in an electrolysis cell, wherein the organic compound is both oxidized and reduced at one electrode.
- 10 2. A process as claimed in claim 1, wherein the organic compound is both oxidized and reduced, in particular hydrogenated, at the anode.
- 3. A process as claimed in claim 1 or 2, wherein the electrode is in contact with at least one hydrogenation catalyst, in particular a noble metal.
- 4. A process as claimed in claim 3, wherein the hydrogenation catalyst, in particular the noble metal, is applied to a graphite felt.
- A process as claimed in claim 3, wherein the hydrogenation catalyst is deposited on the anode
 from suspension.
 - 6. A process as claimed in claim 3, wherein the hydrogenation catalyst in the form of a suspension is brought into contact with the anode.

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7. A process as claimed in claim 1, wherein the organic compound is both reduced and oxidized, in particular oxygenated, at the cathode.

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- 8. A process as claimed in any of claims 1 to 7, wherein the electrode is a gas diffusion electrode.
- 5 9. A process as claimed in any of claims 1 to 8, wherein the organic compound is furan and/or a furan derivative.

Electrolytic transformation of organic compounds

Abstract

In a process for the electrolytic transformation of at least one organic compound in an electrolysis cell, the organic compound is both oxidized and reduced at one electrode.

Declaration, Power of Attorney

Page 1 of 3

0050/050738

We (I), the undersigned inventor(s), hereby declare(s) that:

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Electrolytic transformation of organic compounds

the sp	ecification of which				
	[] is attached hereto.				
	[] was filed on				
	Application Serial No				
	and amended on	•			
	[x] was filed as PCT international application				
	Number PCT/EP00/09135				
	on September 18, 2000				
	and was amended under PCT Article 19				
	on	(if applicable).			

We (I) hereby state that we (I) have reviewed and understand the contents of the above—identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119(a)—(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application(s)

Application No.	Country	Day/Month/Year	Claimed
19944990.2	Germany	20 September 1999	[x] Yes [] No

Declaration

Page 2 of 3

0050/050738

application(s) listed below.		
(Applica	tion Number)	(Filing Date)
(Applica	tion Number)	(Filing Date)
International application desig of this application is not discle first paragraph of 35 U.S.C. § 1	mating the United States, lead in the prior United States, 12, I acknowledge the duty	120 of any United States application(s), or § 365(c) of any PCT listed below and, insofar as the subject matter of each of the claims ates or PCT International application in the manner provided by the to disclose information which is material to patentability as defined ang date of the prior application and the national or PCT International
Application Serial No.	Filing Date	Status (pending, patented, abandoned)
And we (I) hereby appoint:	Norman F. Oblon, Marvin J. Spivak Gregory J. Maier,	Registration Number 24, 618; Registration Number 24, 913; Registration Number 25, 599;
	William E. Beaumont, Steven B. Kelber, Jean-Paul Lavalleye,	
connected therewith; and we (ocation, to prosecute this I) hereby request that all of	application and to transact all business in the Patent Office correspondence regarding this application be sent to the firm of ISTADT, P. C., whose Post Office Address is: Fourth Floor, 1755

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Jefferson Davis Highway, Arlington, Virginia 22202.

Declaration

Page 3 of 3

0050/050738

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February 15, 2002

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